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Geochronology of early human settlements in Java: what is at stake?

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Abstract

Despite the fact Java Island is a volcanic area, it has been challenging to build a chronological framework of its early human settlements, from the oldest Lower Pleistocene *Homo erectus* up to the dispersals that followed the Last Glacial Maximum (LGM). Various dating methods are implemented on volcanic effluents, sedimentary deposits, speleothems and also on fossils (including direct analyses on human remains), thanks to radiometric and palaeodosimetric grounded techniques as well as to magnetostratigraphy. However, a number of results must be considered with caution, in order to identify specific bias originating from reworking of volcanic effluents or post-depositional geochemical evolution. Such bias might well explain apparent contradictions between absolute dating results and other chronologically significant proxies (e.g. biostratigraphy). Converging age estimates resulting from the implementation of several dating methods, though being sometimes less precise, seem to have a higher reliability.

Key-words: *Homo erectus*, ESR/U-series, Ar/Ar, Ngebung, Java, Lower Pleistocene

1. Some critical chapters in human evolution and a favourable geodynamic context

Located on the Sunda shelf, Java Island is part of the Southeast Asian archipelagic area that relates number of critical chapters of the history of human lineage, most of them dealing with the biological and cultural adaptation of humans to a highly changing environmental context during the Quaternary period (see A.-M. Sémah et al.; Grimaud-Hervé et al., in this volume).

Here were found the archaic *Homo erectus* that were the first to become islanders during the Lower Pleistocene (e.g. Sartono & Grimaud-Hervé, 1983). They were followed by more classical forms (e.g. the Trinil *Homo erectus* fossil, Dubois, 1894), that can be the descent of these ancestors and/or new immigrants having followed the opening of dispersal routes from mainland Asia during eustatic drops of the sea level (especially around the Lower to Middle Pleistocene boundary, owing to severe increases in global ice volume records, e.g. see Elderfield et al., 2012). A derived *Homo erectus* group seems to have persisted in the island till the extinction of the taxon (e.g. Oppenoorth, 1931), likely to have occurred during the Upper Pleistocene, and might even have coexisted with the earliest *Homo sapiens* immigrants (Storm et al., 2005). Then it comes with the still poorly documented early history of Anatomically Modern Humans in the archipelagos, whose tiny traces can be followed only in a limited number of sites, predating two major phases of dispersals that are currently the subject of thorough research (see for instance Sémah et al., 2004; Détroit, 2002; Simanjuntak et al., 2014): the expansion of human groups around the Pleistocene-Holocene transition, and subsequently the so-called Austronesian dispersals (see on this topic Bellwood, 2007); Noerwidi, 2012).

Establishing a chronological framework for such a long history may benefit, from several points of view, from a favourable geodynamic context. The Java Island is part of the Sunda inner volcanic arc (e.g. van Bemmelen, 1949; Katili, 1975) and progressively acquired its current morphological features during the Pleistocene. North of a former, Miocene volcanic arc (whose volcanic products can still be observed for instance in the Southern Mountains bordering the coast of the Indian Ocean), huge volcanic cones (still active for part of them) expand along the longitudinal axis of the island. Their effluents represent a productive source of minerals and chemical elements that are useful in geochronology, and allow consider the implementation of various methods, e.g. those grounded on argon or uranium content, or on the magnetic properties of the deposits.

2. A diversity of sites and methods

A variety of sites are found all along the addressed periods, among which we can identify three main categories (Figure 1).

2.1. Sites related to the tectonic evolution of Java Island.

In Central and Eastern Java, many sites are actual structural parts of the island. This is especially the case for the fossil-bearing stratigraphical series that belong either to the central axial basin (the Solo depression, van Bemmelen, *op. cit.*) or to the ridge that borders it to the North (the Kendeng hills). Such sites are historically of major importance.

In the Kendeng hills, they include the Trinil series, excavated by Dubois (*op. cit.*) along the banks of the Solo river, and numerous places that played a major part in the early palaeontological and palaeoanthropological researches carried out before and after Indonesian independence by the Geological Survey (Duyfjes, 1936; van Es, 1931; von Koenigswald, 1949).

Among these sites are those which grounded the vertebrate biostratigraphical frame proposed by von Koenigswald (1949) and subsequently revised (de Vos et al., 1982; 1994), including some that were subject to dating attempts:

- Bumiayu towards the West, including the so-called Satir and Ci Saat faunas that postdate the early Pleistocene recession of the sea from the area, the earliest land mammals being dated to the early normal subchrons of the Matuyama period (Reunion and Olduvai, Sémah, 1986).
- Perning, near Mojokerto, in the Eastern part of the island, where the less pronounced anticlinal folds of the Kendeng hills yielded the skull of the Mojokerto child (see Huffmann et al., 2005) and where ages older than 1.4 Ma were published (Morwood et al., 2003).
- Kedungbrubus, within the Kendeng hills (famous for the find of the so-called Kedungbrubus faunal association dating back to the Middle Pleistocene) near which the Gunung Butak series yielded fossils that count among the oldest in East Java (c. 1.9 Ma, Bandet et al., 1989).

Near Solo town, the Sangiran dome represents the richest hominid-bearing site (von Koenigswald, 1940). Together with other tectonic boutonnières located within the Solo depression that surround the Lawu volcano, and sites belonging to the Southern flank of the

Kendeng hills, it allows to relate the palaeogeographic history of the area (Djubiantono & Sémah, 1993; Sémah, 1986; Watanabe & Kadar, 1985).

The major features of the evolution of the area were first dated using palaeomagnetism: early signs of the recession of the sea following the Gauss-Matuyama boundary, ponding up of the shallow marine basin during and just after the Olduvai subchron; surrection and subsequent erosion of the Kendeng hills northward and of the Southern Mountains around the end of the Lower Pleistocene (before the Matuyama-Brunhes boundary).

This framework was subsequently refined thanks to the implementation of various methods. The latter include argon analyses bearing on pumices, on isolated amphibole grains recovered from tuffaceous layers, and also fission tracks attempts on zircons. Among the major results (Watanabe & Kadar, 1985; Swisher et al., 1994; Saleki, 1997; Sémah et al., 2000a) are the ages of the earliest *Homo erectus* fossils (c. 1.6 Ma) and some other dates obtained in the upper half of the so-called Pucangan (or Sangiran) layers, tentatively correlated with the Ci Saat faunal association (c. 1.2 Ma, de Vos et al., 1994).

Other methods were used as well. Among them is worth to mention the specific pluri-method approach carried out on the Ngebung site (northwestern part of the Sangiran dome, lower so-called Kabuh –or Bapang- series; Saleki, *op. cit*.; Sémah et al., 2011, Fig. 2, Tab.1 and Tab.2), that documents a *Homo erectus* occupation floor where Acheulean like tools were characterized for the first time in Sangiran (Sémah et al., 1992). Whole rock and single grain argon dating of volcanic effluents, ESR dating of volcanic quartz, combined U-series and ESR dating of enamel from herbivorous fossil teeth converge to assign the site an age of 0.8 Ma. Associated to a fauna described as a “late Trinil” association, this site is likely to picture the early exchanges with the mainland that took place just after the installation of Mid-Pleistocene climatic conditions (opening of the landscape and related circulation corridors on the Sunda shelf, see A.-M. Sémah et al., *op. cit*.).

Though a debate is currently open regarding the age of the early fluvio-volcanic layers in certain parts of the dome (Larick et al. 2001), where older ages might be considered as well (see below, Part 3.).

2.2. River terraces

Other sites are related to the geomorphological evolution of the landscape after the major volcanic and tectonic phases. We find in this category the sites related to the river terraces systems of the large Solo river, and to the discoveries of the most derived *Homo erectus* fossils in Ngandong, Sambungmacan and Ngawi (see for instance Oppenoorth, 1931; Jacob et al., 1978; Sartono, 1990).

Somewhat paradoxically, those sites count among the earliest discoveries but still pose a clear problem regarding their age. ESR, combined U-series and ESR dating on fossil teeth enamel assign them an age ranging between the Middle to Upper Pleistocene transition and the Late Pleistocene (Swisher et al., 1996; Falguères et al., 2001 & Fig. 3). Together with direct gamma ray spectrometry carried out on original human fossils (Yokoyama et al., 2008) they even allow to consider the possibility of quite late extinction of *Homo erectus* and of the coexistence with the early *Homo sapiens* that reached the island. However, the latter hypothesis seems hardly compatible with the apparent faunal register that considerably differs from the rain forest adapted Punung fauna (discovered in the Southern Mountains and dated to early OIS 5 stage, see below Part. 2.3.).

2.3. Karstic sites

The third major category is linked to the karstic evolution of limestone massifs. The concerned area includes the cavities that formed in the Miocene limestones of the Southern Mountains, especially in the *Gunung Sewu* near Punung. Known for long for its quite recent archaeological register (e.g. van Heekeren, 1957), the area became famous when von Koenigswald (1936; see also Teilhard de Chardin, 1937) discovered the Palaeolithic Pacitan industry in the alluvia of the Baksoka river. Dating attempts on such alluvial deposits used ESR on bleached quartz grains, and provisional results assign ages ranging between 200 and 300 ka to the 12 m Kiut terrace that contains a lithic industry comparable to the Song Terus late Middle Pleistocene one (see below).

The karstic fillings offer two kinds of deposits:

- Several fissures contain fossil accumulations due to porcupine activity (a common occurrence in mainland and island Southeast Asia) that were described as the Punung fauna association and include taxa well adapted to the rain forest (Badoux, 1959; A.-M. Sémah et al., in this volume), most often cemented within stalagmitic concretions. U-series and OSL dating (Westaway et al., 2007; Tu, 2012) assign an early OIS 5 age to these fossils, in accordance with the palaeoenvironmental record.
- Cave fillings proved to cover a much longer time range than previously supposed by archaeological surveys. As for instance, more than 15 m thick sedimentary series were documented in caves such as Song Terus and Tabuhan (Sémah et al., 2004; Hameau et

al., 2007). Various methods were implemented there, covering the earliest stalagmitic floors (> 350 ka, U-series, Sémah et al., 2000b), the oldest lithic flake assemblages (c. 300 ka, combined U-series and ESR dating of enamel of fossil teeth), and the earliest cave occupation floors (120 to 85 ka, U-series dating of mushroom like stalagmites). Knowing the palaeoenvironmental context (A.-M. Sémah et al., *op. cit*) these ages fit with the dates published regarding the Punung faunal association. Analyses carried out on Late Pleistocene layers, resulting into ages ranging between c. 60 and 30 ka, are more difficult from the dosimetry point of view (see Part 3. below). The uppermost parts of the cave fillings reflect the intensive occupation of the cavities by modern humans during the first half of the Holocene, and current ^{14}C dating range between 9 and 5 ka.

3. Possible bias and current orientations

Among the above mentioned results, several current debates develop, which could have a critical incidence on the overall chronological framework of the early human settlements on Java Island. Some of these issues might be related to a relative lack of precise stratigraphical control or description of the context. Such a situation might be summoned in the case of some samples originating from ancient collections coming from the Solo terraces late *Homo erectus* sites, in order to explain some discrepancies between various studies. But others imply to consider with caution the dating approach, and try to identify both contextual and analytical bias. Among the latter are worth to mention several examples:

3.1. Regarding argon methods implementation

In volcanic areas, argon methods are usually available for building a reliable chronological frame (for details on the dating method, see Renne et al., 1998; Renne et al., 2009; Nomade et al., 2011). Some dates much older than 1 Ma were obtained (Larick et al., 2001) in the so-called Kabuh or Bapang series of the Sangiran dome. Knowing the geographical extension of the site (> 50 sq. km) and the difficulties to establish reliable horizontal correlations, the hypothesis of the existence of such old volcano-sedimentary layers cannot be a priori discarded. Though, it appears that grounding an age assumption only by dating grains extracted from smaller pumice blocks might not fulfil the need of an accurate chronological attempt. As for example, one of these analysed on the Ngebung site (see Part 2.1.) yielded a date of c.1.5 Ma, among numerous samples which point to a 0.8-0.9 Ma age. The Table 1 shows that Argon/Argon dating analyses were performed on 4 pumice and three tuf samples. Small grains of amphiboles and biotite extracted from ash layers in the upper part of Ngebung stratigraphy and in pumice coming from B and C layers were analysed in the Geosciences Azur Laboratory of Nice, France, in the frame of the PhD of H. Saleki (1997) in which the analytic protocole and measurements are described. For pumice sample NG91-4, 4 analyses were performed with increasing the step number for each one. The weighted mean age is about 0.86 ± 0.03 Ma with 2 sigma error range. Three tuff samples were analysed also with two steps for each one. Two samples yield a weighted mean age of 0.90 ± 0.06 Ma that are in agreement with those of pumice layers. The results obtained at Ngebung layers are centred around 0.88 Ma.

While waiting for more complete results obtained throughout the Sangiran area, we must provisionally consider the probability of a natural reworking of some dated samples (i.e. those yielding ages around 1.5 Ma), owing for instance to natural sweeping of volcanic chimneys, or to erosion and re-deposition of volcanic tuff layers that are not fully characterized from the sedimentary dynamics point of view.

3.2. Regarding U-series and combined U-series and ESR dating of fossils, with special reference to the Ngebung site (Sangiran Dome)

In Java, and especially in volcanic context, dosimetry of the embedding sediment is an important parameter in order to interpret the analyses in terms of ages. As for instance in cave deposits, the heterogeneity of the filling heavily influences the final results. This was well demonstrated (Hameau, 2004) in the case of the late Pleistocene layers of the Song Terus cave. Various modelling attempts were carried out, grounding on in situ dosimetry and measurement of samples in the laboratory, in order to consider the respective influence of various kinds of matrix near the collected samples (volcanic ash layers, limestone blocks, karstic clays, other fossils etc.), within the range of reach of the gamma rays (c. 30 cm).

The Ngebung record is of utmost importance for the understanding of *Homo erectus* settlements in the islands at the dawn of the Middle Pleistocene. It pictures the progressive set up of quite new climatic and environmental conditions (Sémah et al., 2010) (increased seasonality, severe fragmentation of the forest, new mammal immigrants) and reflects the severe palaeogeographical changes that occurred on the Sunda shelf.

These independently obtained results are significantly convergent and assign the site an Early Middle Pleistocene age, c. 800 ka. Though, the faunal record doesn't document as yet all the immigrants that reached Java during that period, e.g. *Elephas* (only *Stegodon* is present).

Current excavations and associated dating attempts in other parts of the dome, where this taxon was found, will refine the biostratigraphical, palaeo-anthropological, archaeological and palaeoenvironmental framework of the Sangiran dome.

The Ngebung site offers the possibility to have a multi-proxy approach using also combined U-series (see details in Ivanovich and Harmon, 1992) and ESR methods (Falguères and Bahain, 2002; Grün, 1989; Ikeya, 1993) on herbivorous teeth, ESR on volcanic quartz (Falguères et al., 1994). ESR-US method depends on the mode of uranium uptake in fossil teeth, which can be described by mathematical models; in most cases, it is comprised between the early uptake (EU) and the linear uptake (LU) models. The former assumes that uranium was incorporated shortly after burial (Bischoff and Rosenbauer, 1981), while the latter considers a constant rate of uranium uptake since the time of burial (Ikeya, 1982). The combined ESR/U-series method allows the calculation of the history of uranium uptake using the uptake parameter p (Grün et al., 1988).

Some interesting comparisons were carried out (Saleki, 1997) between U-series and combined U-series and ESR dating on large herbivores fossil teeth and bones. While several consistent dates could be obtained by means of the second approach, classical U-series gave a much younger, c. 100-150 ka age: such an observation might be related to a significant and quite late phase of uranium uptake, that might well be due to a resuming of water percolation within the sandy matrix following the shaping up of the dome, much after the deposition of the layers.

More recently, five herbivore teeth collected from archaeological Ensembles A (NG9801, 02, 03) and B (NG9804 and 05) were analysed using the combined ESR/U-series method calculating a p-value corresponding to the uranium uptake in the different tissues constituting a tooth. All the samples were in relatively good state of preservation. The various tissues were separated mechanically. The enamel was ground, sieved (100-200µm) and split into 10 aliquots for irradiation, after the cleaning of its outer surface to remove the effect of external alpha radiation. No cementum layer was observed and the age calculation was carried out following the dentine-enamel-sediment model (see table 2).

The enamel samples were irradiated with doses of 52, 98, 200, 400, 770, 1830, 2740, 5460 and 9150 Gy, using a calibrated ^{60}Co gamma-ray source. The ESR measurements were performed at room temperature on a Varian E-109 X-band spectrometer, with a microwave power of 10 mW, and amplitude modulation of 1 G (0.1 mT). A scan range of 100 G (10 mT) and a time constant of 4 minutes with a modulation frequency of 100 KHz were used for each spectrum. The equivalent doses (D_E) were determined from the asymmetric enamel signal at g=2.0018 by the additive method, using an exponential fitting (Yokoyama et al., 1985). Each ESR measurement was repeated three times for each dose.

U-series analyses were made on each dental tissue, using alpha-ray spectrometry and according to the standard methods described by Bischoff et al. (1988), at the Department of Prehistory of Muséum national d'Histoire naturelle, Paris.

ESR age calculations were carried out by the ESR-DATA program (Grün, 2009) using an alpha efficiency of 0.13 ± 0.20 (Grün and Katzenberger-Apel, 1994) and Monte-Carlo beta attenuation factors (Marsh, 1999), based on the thickness of the tooth enamel and of the removed outer layers. In addition, the following parameters were used:

- Water content was estimated to be 3 wt% in the enamel, 10 wt% in the dentine and 22 wt% in the sediment, based on the wet weight.
- Gamma-ray spectrometry was used to determine the radioisotope (U, Th and K) content of the sediments where the teeth were collected; the dose rate was calculated according to Adamiec and Aitken (1998).
- The effect of Rn loss in each tissue was determined by combining alpha-ray and gamma-ray data (Bahain et al., 1992).

Quartz grains ESR analyses (500 µm fraction) used the aluminium centre. The samples were irradiated at various doses from 100 to 12000 Gy, with a calibrated ^{60}Co gamma-ray source (CEA, Saclay, France). The ESR measurements were performed at 105K on a Bruker EMX X-band spectrometer, each aliquot being measured two times after one rotation of the tube in the ESR cavity. The equivalent doses (D_E) were determined from ESR intensities versus dose growth curve with an exponential fitting function using Microcal OriginPro 8 software with 1/I2 weighting (see details in Voinchet et al., 2013). The annual dose was calculated from the U, Th, K content in the sediments. The beta dose was calculated with an attenuation factor of 0.556 (Brennan, 2003). The cosmic dose was determined from the thickness of the layers according Prescott et al., 1994. An internal dose of 100 microGy, representing about 10% of the total annual dose, was estimated.

4. Conclusion

Studies regarding geochronology of the Quaternary period in Java are still under development, and include interesting issues like those which arise from the implementation of recently developed techniques in U-series, such as the recently revised age of the Wajak fossil (Storm et al., 2013) that opens new perspectives regarding the LGM to Holocene faunal environment evolution (see Amano et al., in this volume).

Dating the palaeontological, archaeological and palaeoanthropological record of Java is therefore a critical but complex task. Experience shows that converging age estimates resulting from the implementation of several dating methods, though being sometimes less precise, have a higher reliability and constitute the best way in order to solve numerous issues, while looking forward the concomitant implementation of new approaches such as ESR dating on shells and diatomitic beds, or cosmogenic nuclids dating on detritic siliceous rocks.

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FIGURES AND TABLE

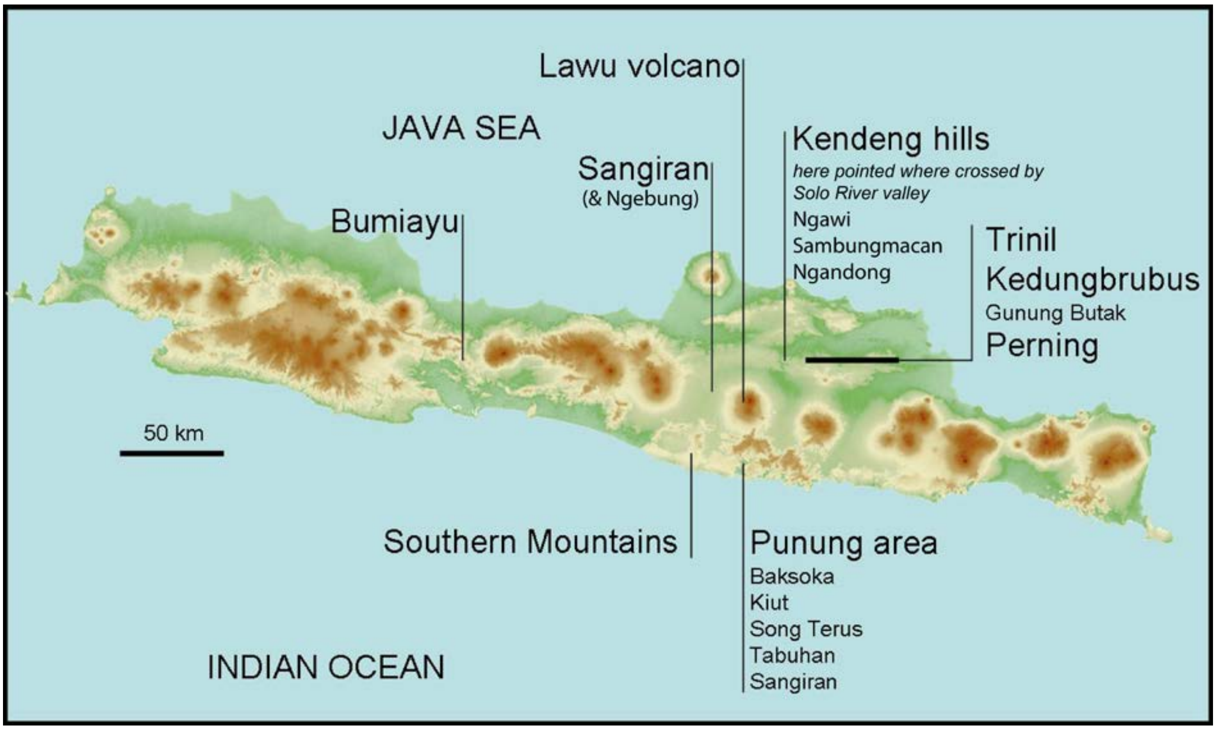
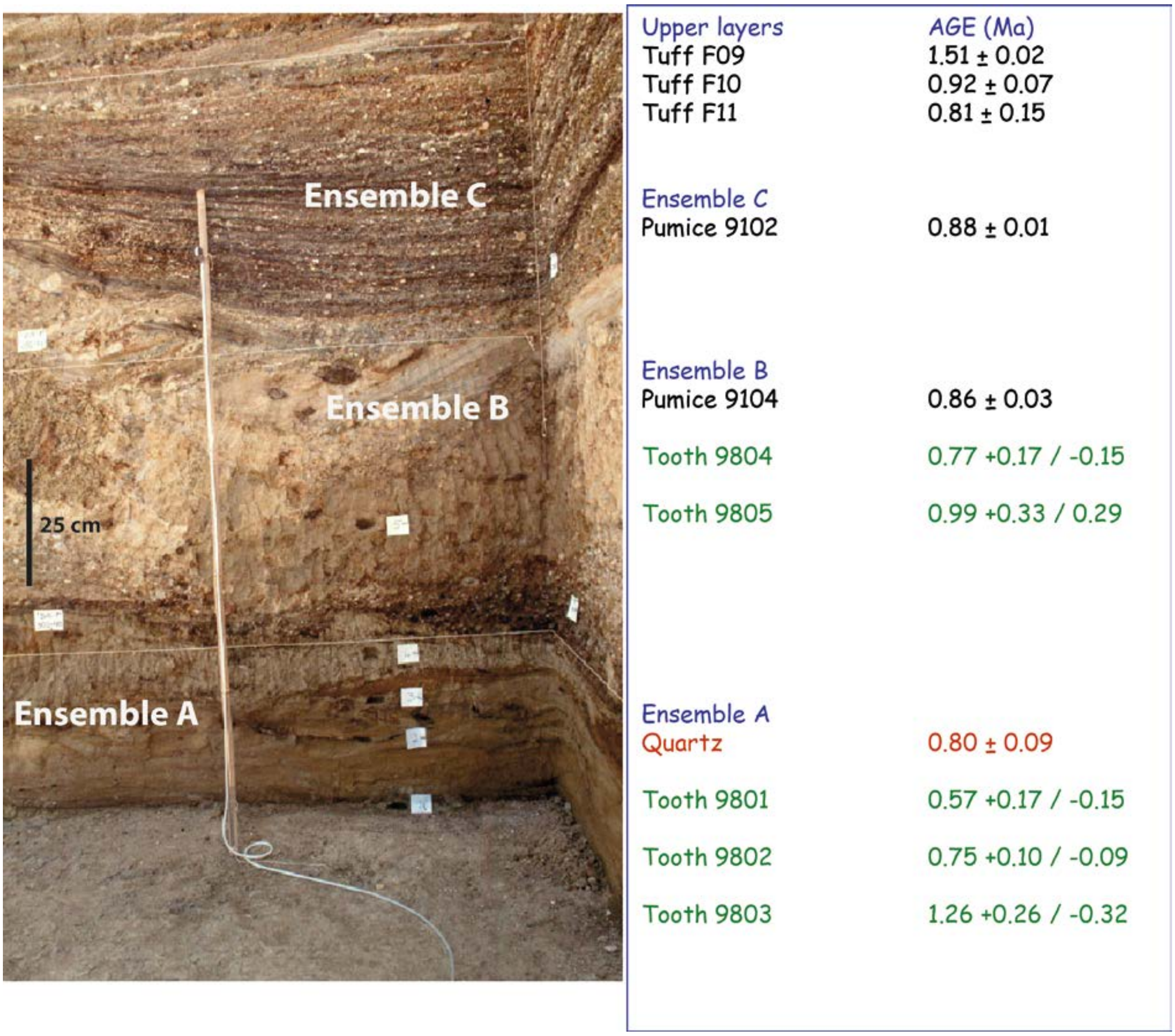


Fig. 1. Map of Java Island with location of the quoted sites



Upper layers	AGE (Ma)
Tuff F09	1.51 ± 0.02
Tuff F10	0.92 ± 0.07
Tuff F11	0.81 ± 0.15
Ensemble C	
Pumice 9102	0.88 ± 0.01
Ensemble B	
Pumice 9104	0.86 ± 0.03
Tooth 9804	0.77 +0.17 / -0.15
Tooth 9805	0.99 +0.33 / 0.29
Ensemble A	
Quartz	0.80 ± 0.09
Tooth 9801	0.57 +0.17 / -0.15
Tooth 9802	0.75 +0.10 / -0.09
Tooth 9803	1.26 +0.26 / -0.32

Fig. 2. The Ngebung site (Sangiran, Central Java) dating attempts (after Sémah et al., 2011)

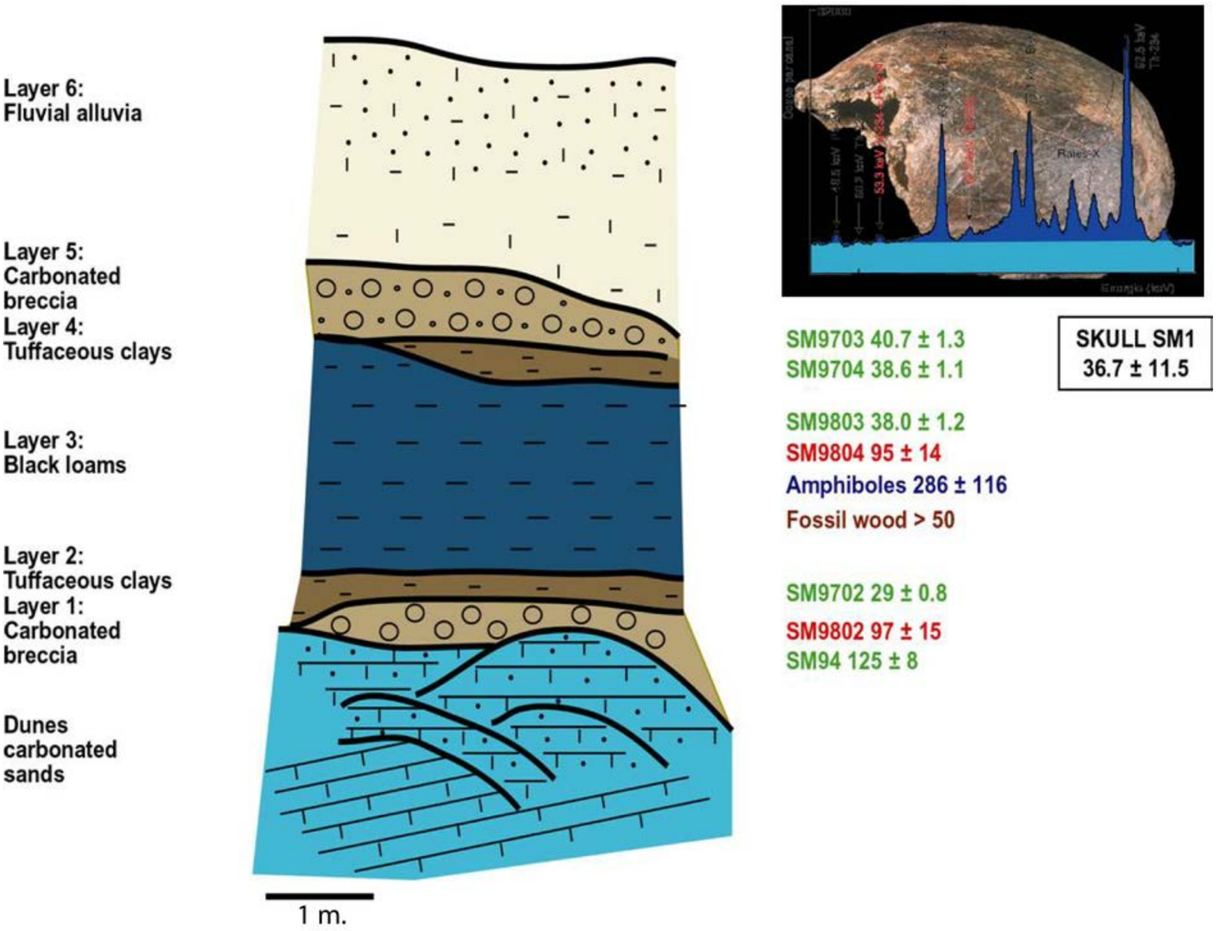


Fig. 3. The Sambungmacan site (Central Java, after Falguères et al., 2001) dating attempts: in red, ESR/U-series on herbivorous teeth; in green, U-series on animal bones; in brown, 14C on fossil wood, in blue, Ar/Ar on amphiboles, in black U-series using non destructive gamma-ray spectrometry on the human skull SM1 published in Yokoyama et al., 2008.

Samples	Units	Tissue	U (ppm)	(ß + γ) sediment + cosmic (µGy / a)	Internal dose rate (α+ß) enamel (µGy / a)	Total dose rate (µGy / a)	DE (Gy)	p enamel	p dentine	Age (ka)
NG9804	**B**	E	5	491	1497	1988	1530 ± 150	-0,6 ± 0,3	0,03 ± 0,3	770+172/-152
		D	87							
NG9805		E	16	476	1036	1512	1510 ± 150	3,7 ± 1,8	1,2 ± 0,8	999+326/-289
		D	101							
NGQuartz				1185	100	1285	1023 ± 53			796 ± 90
NG9801	**A**	E	64	480	10603	11083	6270 ± 600	-0,03 ± 0,4	-0,8 ± 0,1	566+170/-151
		D	258							
NG9802		E	12	485	4888	5373	4000 ± 400	-0,7 ± 0,1	-0,6 ± 0,1	745+103/-87
		D	137							
NG9803		E	10	504	808	1312	1650 ± 160	11,3 ± 2,4	1,6 ± 1,0	1258+256/-322
		D	271							

Table 1: Components of dose-rates for US model of teeth and sediment, and combined ESR/U-series age estimates with correspondent p-values for fossil teeth from Ngebung. External dose-rates correspond to both sediment dose and cosmic dose (ß + γ). Two types of measurements have been performed. About 100g of sediment including rock fragments when they are present, were measured at least one month after it has been inserted in a box. Sediments have been sampled in the same square and at the same height as the teeth. TL dosimeters have been placed in different levels at the exact location of the analyzed sediments. All samples lack cementum tissue implying that the enamel was directly in contact with the sediment. The case dentine-enamel-sediment was systematically used. For quartz, the beta dose was calculated with an attenuation factor of 0.556 (Brennan, 2003).

sample	number of grains	Step	39Ar %	Atmos Ar %	37ArCa/39ArK	40Ar*/39ArK	AGE (Ma)
Tuf F09 (V308)	4	2	0.053	97.2	2.64	1.58 ± 5.2	3.34 ± 11.2
			99.947	5.0	3.79	0.716 ± 0.011	**1.51 ± 0.02**
Tuf F10 (V367)	4	2	0.862	99	1.76	0.38 ± 0.033	0.79 ± 3.82
			99.138	71.7	7.21	0.435 ± 0.033	**0.92 ± 0.07**
Tuf F11 (V310)	5	2	0.086	100	7.3	-	-
			99.914	96.3	7.56	0.382 ± 0.071	**0.81 ± 0.15**

Pumice NG91-4 (M913)	2	2	0.463	100	4.223	-	-
			99.537	8.86	3.275	0.436 ± 0.02	**0.95 ± 0.05**
Pumice NG91-4 (M911)	1	1	100	25.4	3.34	0.427 ± 0.05	**0.93 ± 0.11**
Pumice NG91-4 (M834)	1	7	0.155	97.13	9.429	2.690 ± 5.00	5.83 ± 10.82
			0.223	74.60	9.052	0.809 ± 2.99	1.76 ± 6.49
			0.280	100	6.798	-	-
			0.364	100	4.101	-	-
			4.187	67.45	3.763	0.202 ± 0.217	0.44 ± 0.47
			19.992	36.35	3.600	0.357 ± 0.05	**0.78 ± 0.11**
			74.799	50.51	3.550	0.394 ± 0.02	**0.86 ± 0.04**
Pumice NG91-4 (M836)	1	3	0.541	100	6.25	-	-
			4.072	63.87	3.21	0.209 ± 0.352	0.45 ± 0.76
			95.387	64.72	3.12	0.360 ± 0.021	**0.78 ± 0.05**

Table 2. ^{40}Ar/^{39}Ar data obtained on single grains. The error bars are given at the one sigma level. The laboratory experiment numbers are given in parentheses. ^{40}Ar*=radiogenic ^{40}Ar